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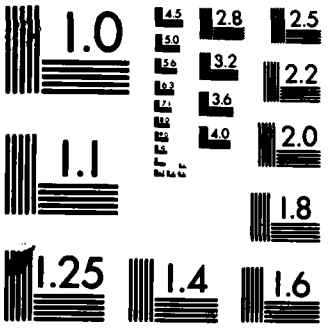

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Creep of Carbon Yarns and Composites at High Temperature

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John A. Criscuolo, Major, USAF
Project Officer

Joseph Hess, GM-15, Director
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Creep of mesophase- and rayon-based carbon yarns was measured in 2 to 4 ksi tension from 2100°C to failure (2500-2700°C). Resulting creep rates increased with temperature, and activation energies were lower for mesophase- than for rayon-based yarn. Rates of creep measured in pitch-matrix-impregnated yarn bundles were lower than those for unimpregnated bundles.

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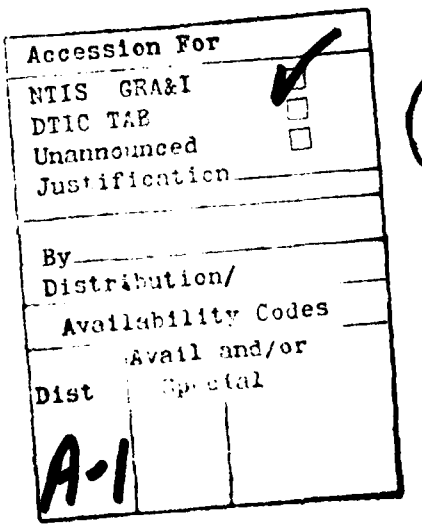
PREFACE

This work was supported by the Office of Naval Research under the direction of Dr. L. H. Peebles (Contract No. F04701-83-C-0084). The author thanks Dr. R. A. Meyer for initiating this effort and Dr. P. J. Blatz and Prof. G. A. Sines for helpful discussions. The valuable experimental assistance of D. C. Robinson is gratefully acknowledged.



CONTENTS

FIGURES

I. INTRODUCTION

Processing of carbon-carbon composites is affected both by the stresses that occur during pyrolysis and by the thermal expansion anisotropy of the reinforcing carbon fibers and matrix materials. Defects, such as delaminations in two-dimensional composites and cracks and debonds in three-dimensional composites, appear in the final structure. As the material is heated above 2000°C during processing, stresses begin to relax by means of creep (time-dependent deformation). The effects of creep on the stresses developed later on cooling have not been fully investigated, in part because of a lack of information about the creep behavior of the load-bearing fibers during composite processing. Some information has been presented in the literature on creep behavior of carbon fibers.[1-4] However, the modeling of composite behavior during processing has been based mainly on thermal expansion and elastic and fracture properties.[5] The high-temperature creep behavior of some carbon fibers used in carbon-carbon composites is investigated here.

II. EXPERIMENTAL

Creep experiments were conducted using two types of Union Carbide carbon fibers in the form of multifilament yarns: VSB-32 (or P55), a mesophase-pitch-based carbon fiber consisting of 2000 filaments (10 μm average diameter) per yarn with nominal Young's modulus of 55 Msi; and WCA carbon cloth, from which continuous sections of yarn were removed. Also tested were simple carbon-carbon composite samples produced from yarns impregnated at 400°C with mesophase-transformed A240 pitch and calcined at 800°C under argon. A scanning electron microscope (SEM) photograph of a typical section of impregnated P55 yarn is presented in Fig. 1. Creep was measured at increasing temperatures above 2000°C, using a horizontal graphite tube furnace. As indicated in Fig. 2, the yarn sample was passed through the tube, after one end was anchored externally. The other sample end was attached to a flexible wire passing over a pulley and connected to a hanging weight at the other end. The experiment was conducted in an inert argon atmosphere inside the furnace. Displacement of the fiber end was detected by a linear variable differential transformer (LVDT). A feedback controller and automatic optical pyrometer regulated the temperature, and a digital data logger recorded data. Scanning electron microscopy, X-ray diffraction, and electrical resistance measurements also were used to determine the changes in fibers caused by high-temperature creep.

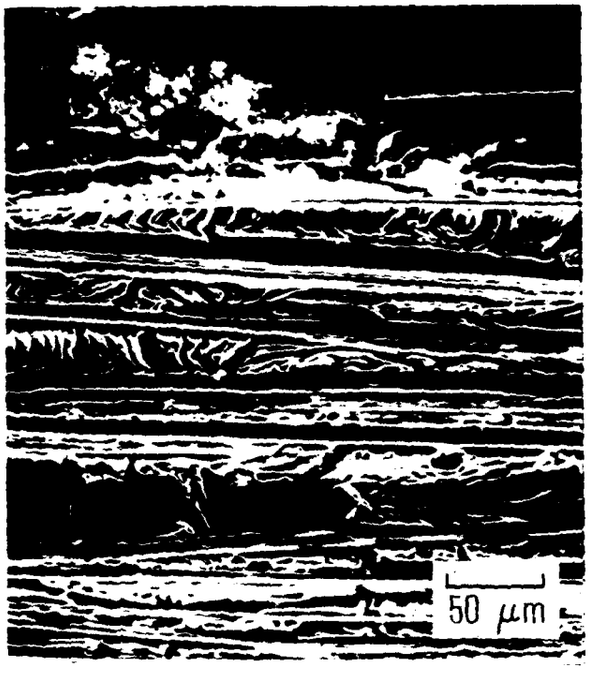


Fig. 1. SEM view of mesophase-pitch-impregnated P55 yarn after 2750°C under load.

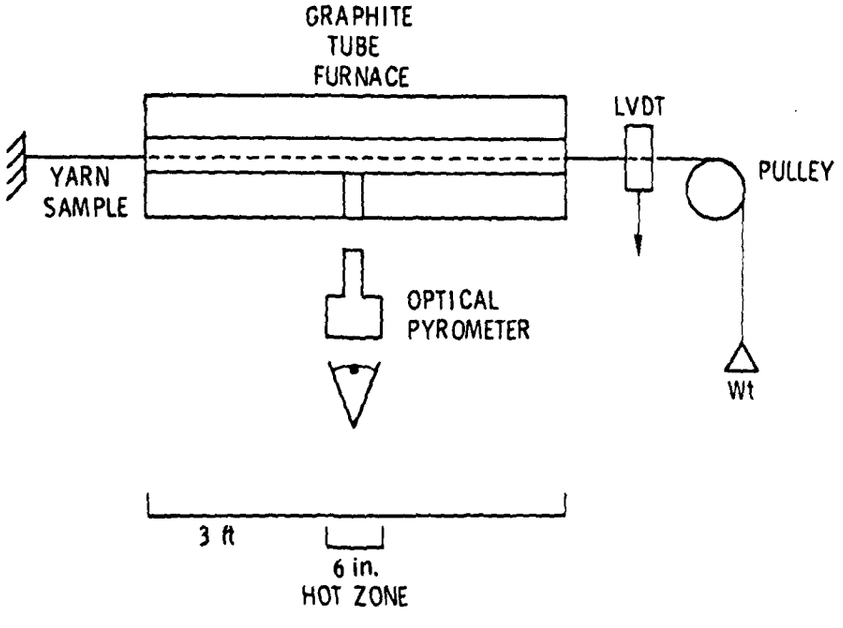


Fig. 2. Schematic of carbon-yarn creep experiment.

III. RESULTS AND DISCUSSION

A typical experimental result for creep, Fig. 3, indicates that creep rate increases steadily with temperature from 2060 to 2600°C, then necks down at 2600°C, as the inset illustrates for a WCA filament. Diameter reduction is greater than a factor of 3. Filaments became extremely narrow (0.1-0.5 μm) near the point of failure, separating into subfilaments over a region 50 to 100 μm in length (original fiber diameter ~ 10 μm) before the actual point of failure. A comparison of the failed ends of dry P55 filaments with pitch-matrix-impregnated P55 filaments, Fig. 4, reveals that the impregnated filaments have narrowed slightly (to ~ 6-7 μm) but have fractured ends with a rough, serrated appearance.

The observed creep strain rates were influenced by the heat-treatment history. Figure 5 compares typical elongation rates above 2000°C for P55 yarns, both as-received and heat-treated, and WCA yarn. The indicated elongation rates are significantly higher for the P55 as-received than the same material after heat treatment at 2600°C, which is consistent with the increase in graphitization of the material at high temperature. The elongation rate of the WCA yarn falls between the two P55 results. This result agrees with the fact that WCA cloth is carbonized from rayon cloth at high temperatures, so that further graphitization and hot-stretching in the experiment are not expected below the previous graphitization temperature.

An Arrhenius plot of log of elongation rate versus inverse temperature is roughly linear for the WCA and P55 dry yarns. The steady-state creep rate is typically given by a relation of the form[2]

$$\dot{\varepsilon} = A \exp(-\Delta H/RT) \qquad (1)$$

Average activation energies were 109 and 66 kcal/mol (see Fig. 6), which contrast with the behavior of the impregnated P55 material plotted in Fig. 7. A drop is observed in the elongation rate of the impregnated yarn sample at around 2400°C and may be caused by the continuing graphitization of the matrix

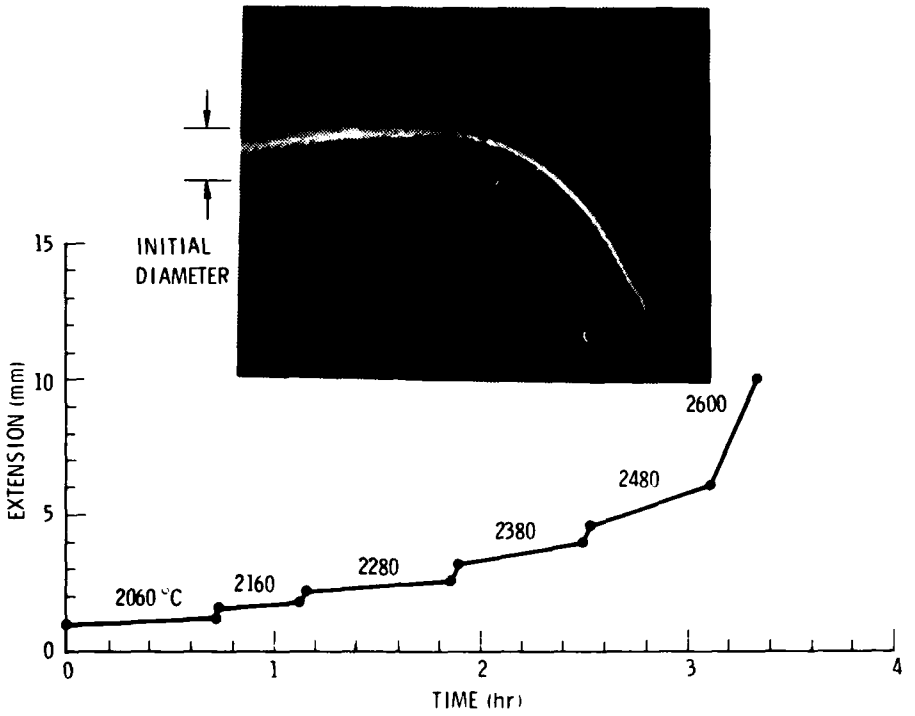


Fig. 3. Typical creep behavior (observed experimentally) of unimpregnated yarn, increasing with temperature. Inset shows failed end of typical WCA filament in hot zone.

Fig. 4. High-temperature failure samples: (a) mesophase-pitch impregnated; (b) P55, dry.

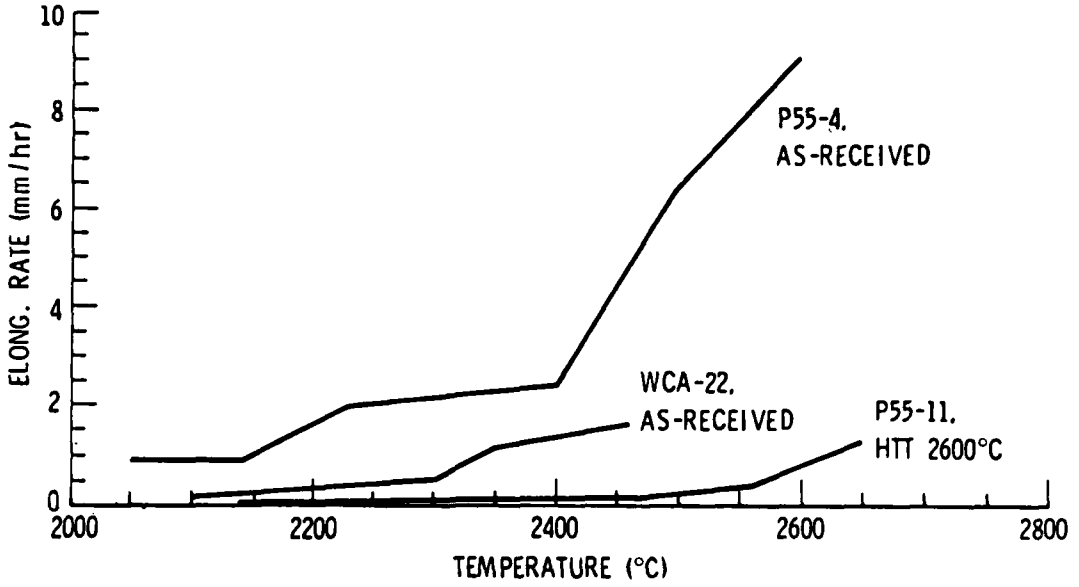


Fig. 5. Comparison of elongation rates for three yarn samples, showing marked difference between heat-treated and untreated P55 yarns.

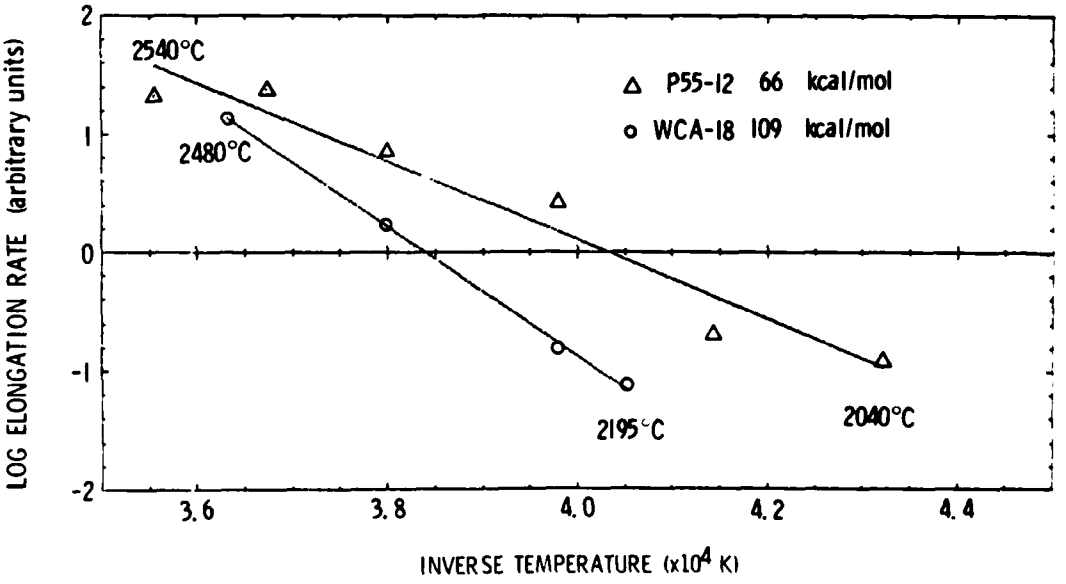


Fig. 6. Thermally activated behavior of dry-yarn creep (elongation) rates.

material and the contrasting thermomechanical properties of the matrix and filaments, though further work is needed to confirm the cause.

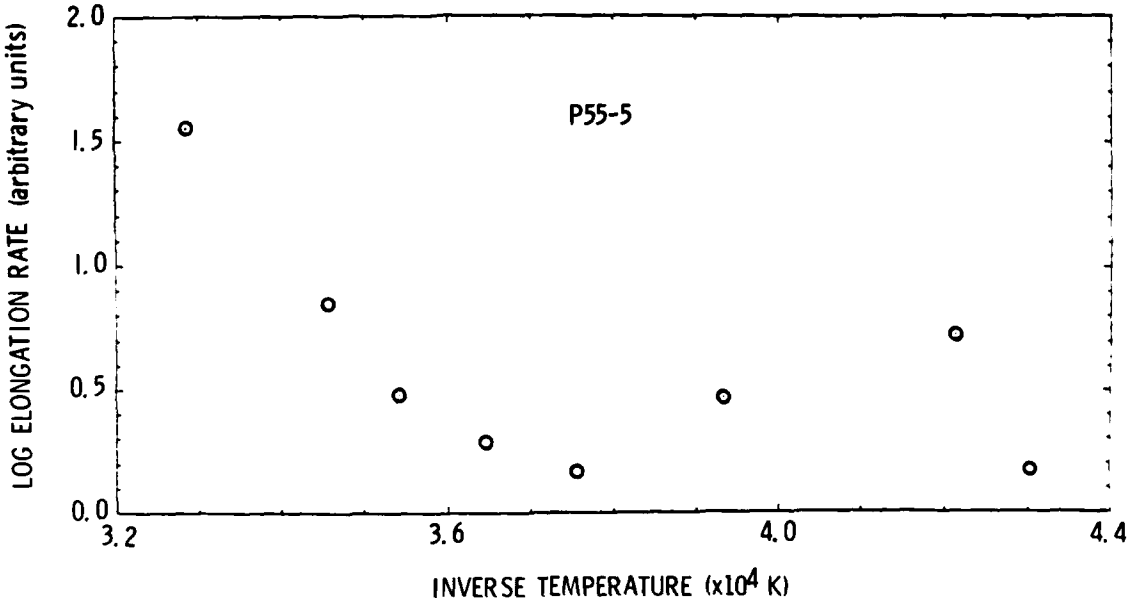


Fig. 7. Temperature dependence of impregnated P55 creep rate, showing marked departure from linearity.

X-ray diffraction revealed slight evidence that graphitization increases during heating, but not as a result of the applied stress. Unstressed fiber samples heated along with creep specimens exhibited similar changes in c-spacing and electrical resistivity. P55 yarn showed a decrease in the d(002) spacing from 0.3447 to 0.3392 nm; the WCA showed a decrease from 0.346 to 0.339 nm. Both c-spacings remained above the ideal graphite value of 0.335 nm.[6] Measurements of the resistivity ratio [ρ(77 K)/ρ(300 K)] increased from 1.12 to 1.67 for P55 after heat treatment at 2500°C, for both unstressed and creep samples. The WCA showed no change in resistivity ratio (less than 1%). X-ray diffraction indicated higher preferred orientation in P55 than WCA but little change in preferred orientation after heating or creep.

IV. CONCLUSIONS

Unimpregnated yarns of rayon-based and mesophase-pitch-based fibers demonstrated basic thermally activated creep behavior, whereas the creep-rate temperature dependence of matrix-impregnated yarns was more complex. Comparison of the failure behavior of samples indicated that necking was substantially less in impregnated than in unimpregnated yarns. Finally, the preferred orientation and crystallinity were altered by treatment at high temperature but were not affected by the degree of creep strain.

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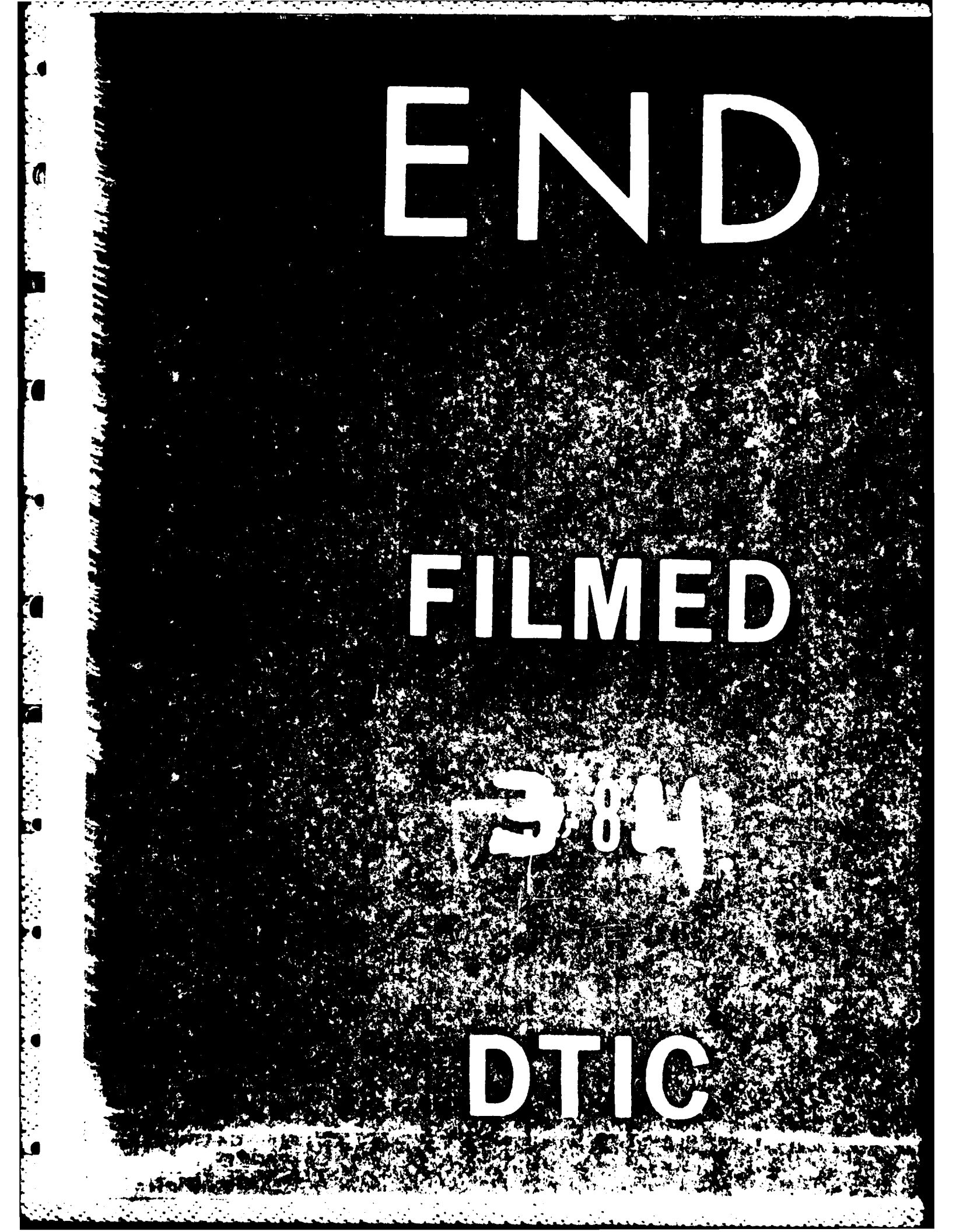
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